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The interaction of recrystallizing interfaces with intragranular precipitate dispersions in a nickel-base superalloy

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This paper is concerned with the ways in which a recrystallizing interface can interact with a dispersion of γ' precipitates in a commercial nickel-base superalloy. It is shown that two major phenomena occur. Firstly, rapid coarsening of the γ' at the interface can lead to the formation of a low number density of large precipitates. Secondly, complete dissolution of the precipitate can occur with subsequent reprecipitation at the interface in a manner which is similar to that of a discontinuous/cellular reaction.

1. Introduction

The microstructures, and hence the properties, of turbine components manufactured from nickel-base superalloys are a sensitive function of many variables in the processing route, as discussed by Bee *et al.* [1]. In powder produced materials, it has been shown that the final structure in finished components produced by thermomechanical processing and subsequent heat treatment is largely determined by the initial microstructure of the compact stock. In particular, the development of the "necklace" structure, [1] which appears to give the optimum compromise in mechanical properties, is governed by the distribution of the γ' phase. A mechanism for the nucleation and growth of this "necklace" structure has already been presented in general terms [1]. However, it is important that the detailed interaction between the recrystallizing interface and the γ' precipitate dispersion is clearly understood.

Many previous investigations have examined the influence of distributions of particles of different sizes on the kinetics of recrystallization [2–6]. However, in some cases the passage of a recrystallization interface through a distribution of precipitate particles causes changes to occur in the distribution itself, for example θ' in Al–Cu [7], NbC in an austenitic stainless steel [8] and γ' in Ni–Al alloys [9].

The present paper discusses recrystallization behaviour in an alloy containing a high volume fraction of large γ' particles. In particular the investigation concentrates on the type of interaction which occurs between recrystallizing interfaces and established γ' particle distributions, and compares and contrasts this behaviour with that reported previously in other nickel-based alloys [9, 10].

2. Experimental procedures

The material used in this study was a powder-produced disc alloy, which has the nominal composition

Co	Cr	Mo	Al	Ti	C	Ni
17	15	5	4	3.5	0.03wt%	balance

This material had been consolidated by hot isostatic pressing (HIPing) at a temperature above the γ' solvus. Billets were thermomechanically processed by isothermal forging to 66% reduction in height at 1050° C, with subsequent heat treatment at 1080° C, (the temperature of the first stage of the standard industrial three stage heat treatment).

Thin foils for transmission electron microscopy (TEM) were prepared by standard techniques and examined in a Philips EM301 machine.

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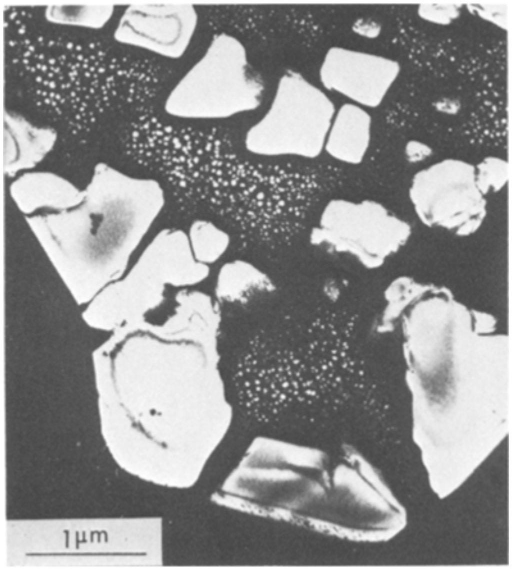

Figure 1 A centred dark field micrograph showing the γ' precipitate distribution in as-HIPed specimen material.

3. Results and discussion

3.1. General

A typical example of the as-HIPed microstructure is shown in Fig. 1. Essentially, the relatively slow cooling from the HIPing temperature leads to the development of massive γ' precipitates on the grain boundaries, while in the grain interiors, a uniform distribution of cuboidal γ' is produced. A fine spherical morphology of γ' is also evident in the intercuboidal regions, although this is not present at the subsequent recrystallization temperature.

Thermomechanical processing of this material produces a heavily dislocated structure in which the cuboidal γ' precipitates are heavily distorted. However, there is little evidence for any change in the spatial or size distribution of the γ' particles, nor of any significant amount of dynamic recrystallization [1] (Fig. 2).

3.2 The effects of a recrystallization anneal

The dramatic effect of heat treatment at 1080° C on the γ' distribution is illustrated in Fig. 3a, in which it can be seen that the original cuboidal array of γ' has been replaced by a small number of massive γ' precipitates. A possible mechanism for the development of this necklace of small, recrystallized grains and its subsequent growth from the original high angle grain boundaries toward the centres of each grain, has already been described [1]. During this recrystallization process, the warm-worked regions develop a fully recovered substructure with the cell size determined by the spatial distribution of cuboidal γ' (Fig. 3b). It is clear therefore that the massive redistribution of solute which occurs during recrystallization is due to the interaction between the recrystallizing interface and the pre-existing

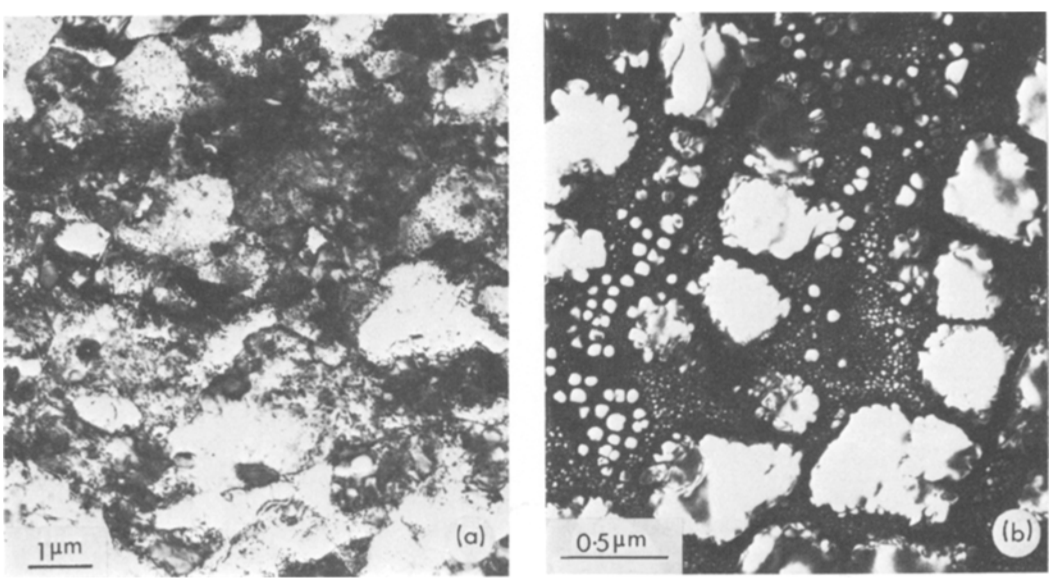

Figure 2 The effect of thermomechanical processing on the as-HIPed structure. (a) A bright field micrograph illustrating the heavily dislocated structure. (b) A γ' centred dark field micrograph. Although highly distorted, the γ' maintains its uniformity of size and spatial distribution.

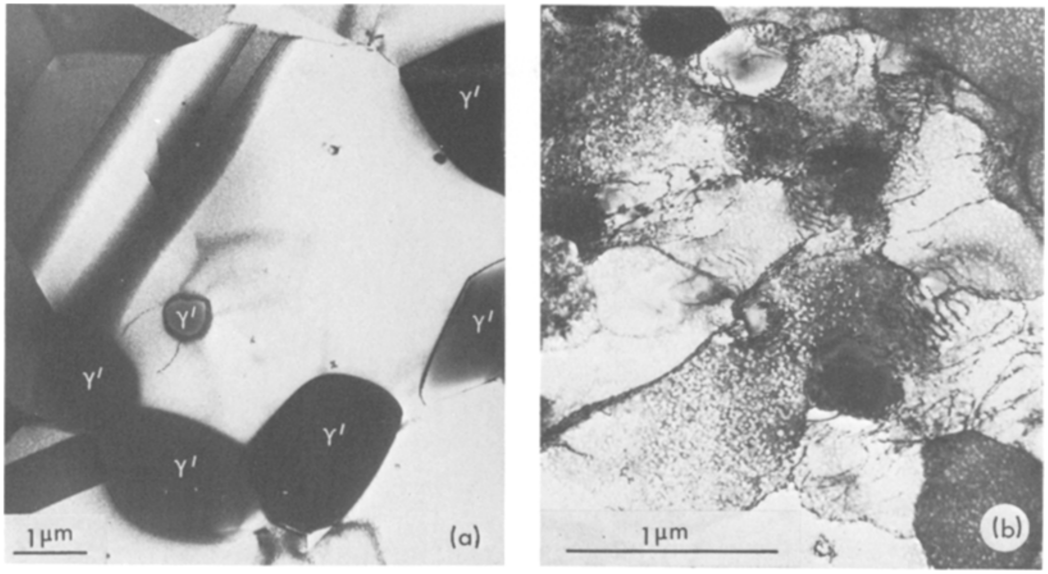

Figure 3 The effect of a recrystallization anneal on the forged structure. (a) Bright field micrograph of a recrystallized region. The uniform γ' dispersion has been replaced by a small number of massive γ' precipitates which are, in general, located at the γ grain boundaries. (b) Bright field micrograph of the warm-worked region. A fully recovered substructure is observed, the cell dimensions being governed by the γ' distribution.

cuboidal γ' dispersion of the recovered substructure.

Observations of the recrystallizing interface support the view that there is exaggerated coarsening of the γ' dispersion at this mobile boundary. This occurs with the concomitant dissolution of the majority of the remaining cuboidal γ' precipitates. Typical examples of this dissolution process are illustrated in Fig. 4a and b. It can be seen that most of the γ' particles in the recovered

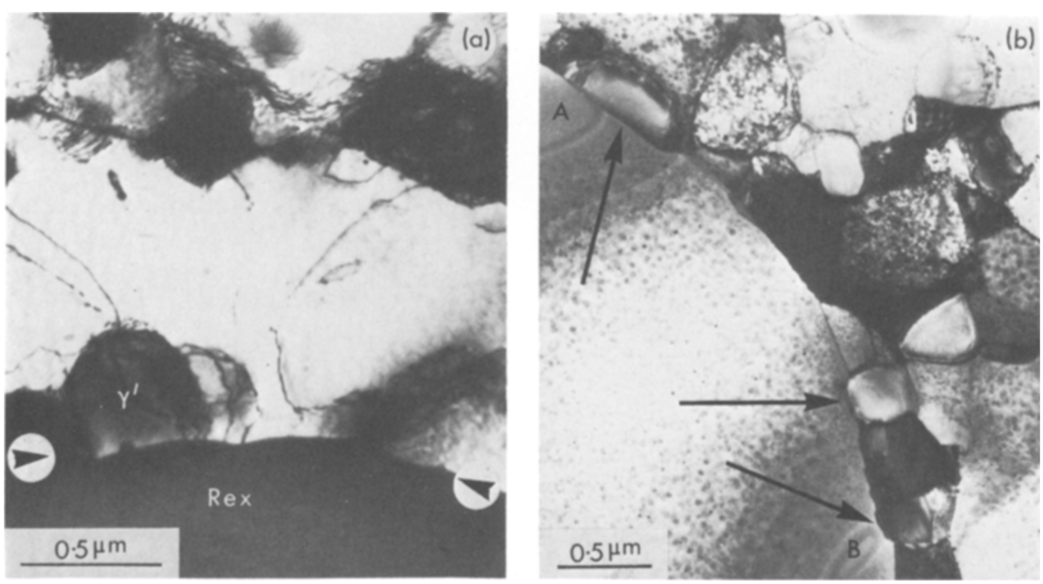

Figure 4 Dissolution of the cuboidal γ' at the advancing recrystallization interface. (a) Bright field micrograph illustrating the dissolution of a single γ' precipitate at the advancing interface (arrowed). (b) Bright field micrograph of a number of dissolving precipitates (arrowed). It can also be seen that a coarse discontinuous reaction is developing in association with the recrystallization front, the two large γ' precipitates (A, B) being in contact with the interface and displaying a cube/cube orientation with the recrystallized grain (see text). The fine spherical γ' observed in the matrix is due to a relatively slow quench from the recrystallization temperature in this specimen. This dispersion is not present during the anneal [1].

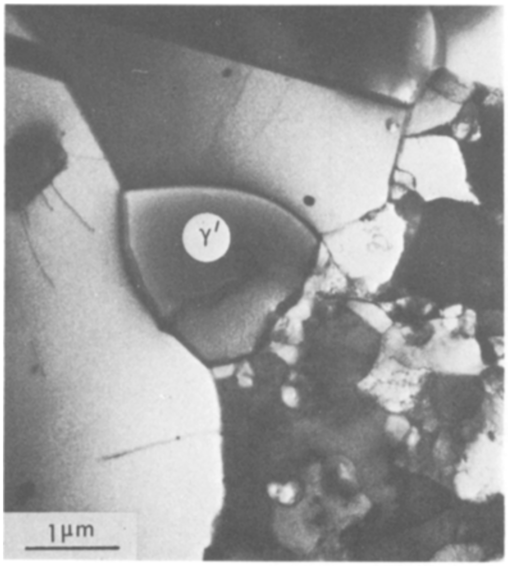

Figure 5 Bright field micrograph of a single γ' precipitate. It can be seen that this particle is pinning the interface. No rational orientation relationship exists between this precipitate and the two abutting recrystallized grains.

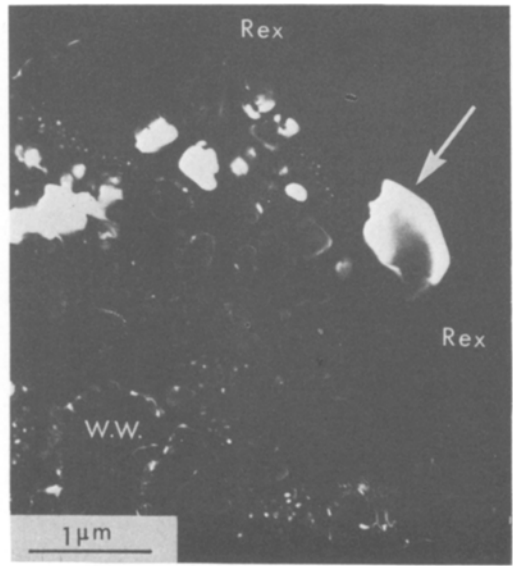

Figure 6 A γ' centred dark field micrograph of a large interfacially coarsened γ' precipitate (arrowed). Much of the γ' in the warm-worked region (W. W.) is also illuminated implying that the interfacial γ' is close in orientation to that in the unrecrystallized area.

(warm-worked) regions are dissolving at the interface, since the arrowed precipitates are not intruding appreciably into the recrystallized grains. There is also little evidence to suggest that there is bowing of the boundary around these precipitates.

Excluding the initial formation of recrystallization nuclei on the large, grain boundary-nucleated γ' (see Bee *et al.* [1]) subsequent development of most of the massive precipitates would seem to rely on the coalescence of some of the clusters of cuboidal γ' which are not dissolved at the recrystallizing interface. An example of a large precipitate in contact with the interface is shown in Fig. 5. Two types of observation support this suggestion that these precipitates originate in the warm-worked regions:

(1) In general, the large γ' particles in the recrystallized areas bear no rational orientation relationship with respect to any abutting recrystallized grain.

(2) It is often found that coarse γ' particles in contact with the mobile boundary are closely orientated with respect to the adjacent warm-worked region (see Fig. 6).

Although most of the precipitate/boundary interactions can be explained in terms of enhanced coarsening processes, there are some isolated, more complex events.

In some instances, reprecipitation is observed on the migrating boundary itself. This process generally occurs in boundary regions where there are no neighbouring large undissolved γ' precipitates. The resultant particles displayed a cube/cube orientation with the associated recrystallized grain. Isolated examples are also found in which more than one of these boundary nucleated γ' precipitates develop with a morphology similar to that of a coarse cellular or discontinuous colony (Fig. 7).

Other observations suggest that the local driving force for migration of the recrystallizing interface can exceed that required to break away from a pinning particle. Some evidence for this is seen in recrystallized grains containing intragranular γ' precipitates (Fig. 8). Since these particles are invariably cube/cube related to the matrix γ grain, they also develop by a reprecipitation process.

The role of the frequently observed annealing twins in the recrystallized regions is not yet clearly understood. However, it would appear that the twinning operation can release a mobile high-angle boundary segment while leaving a residual coherent twin interface. This effectively cuts off the high diffusivity solute path to the precipitate: the precipitate will therefore be by-passed (Fig. 9).

Diffraction analysis of the warm-worked regions reveals large local misorientations. In

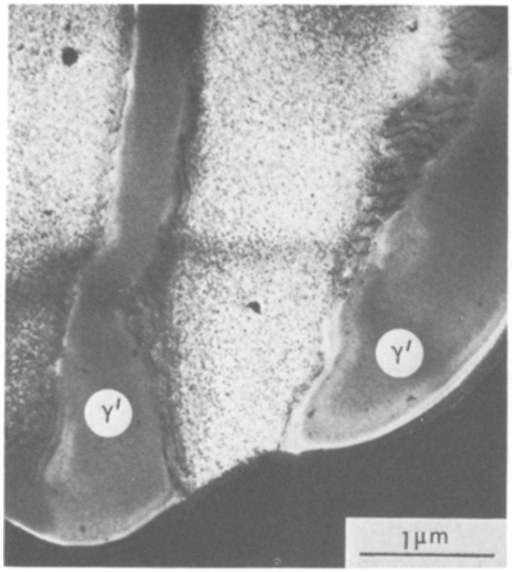

Figure 7 A γ/γ' centred dark field micrograph of a recrystallized grain. This structure is similar to that of a coarse discontinuous colony. The γ' is cube/cube related to the γ.

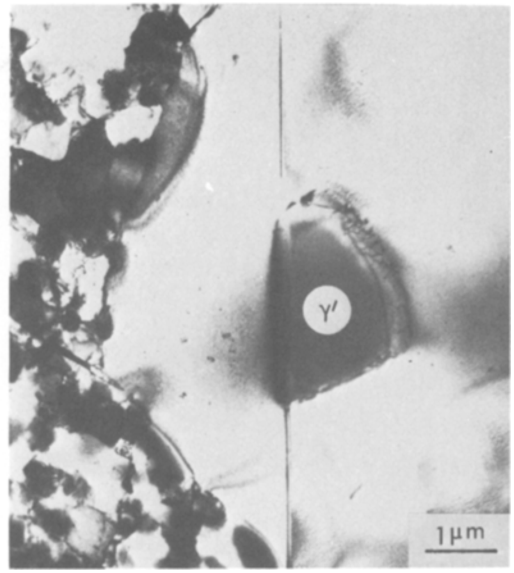

Figure 9 Bright field micrograph of a large γ' precipitate apparently by-passed by a twinning mechanism in close proximity to the recrystallization interface.

particular, a misorientation change of $\sim 30°$ was found in traversing $\sim 7\,\mu$m of a warm-worked region and this was not atypical. This observation is particularly important since it helps to explain the lack of an orientation relationship between large undissolved γ' particles and surrounding recrystallized regions. The observation is also of importance to the proposal that in this material

Figure 8 Bright field micrograph of a large intragranular γ' particle which is cube/cube related to the γ. Note also the interfacial dislocation networks.

recrystallization occurs by continuous renucleation [1], since it means that subgrain coalescence can lead to the formation of new high-angle boundaries only a short distance ahead of a pinned region of a recrystallization interface.

At this point it is useful to compare the present observations with those made by Kreye *et al.* [9] on a cold-rolled and annealed nickel–aluminium alloy. These workers also observed that γ' particles which were present in the deformed regions were dissolved at the recrystallization interface. Furthermore, as in the present investigation the larger particles in the γ' distribution were found to be by-passed, without dissolution at the recrystallization interface.

The observations of the present investigation, however, were also found to differ in some important respects. For the alloys and deformation levels (70% in cold rolling) investigated by Kreye *et al.* particles which were smaller than 60 Å were found to dissolve in contact with the recrystallization interface. In the present investigation particles which were considerably larger than this (up to $\sim 0.5\,\mu$m) were found to dissolve. A second major difference concerns the reprecipitation of γ' observed by Kreye *et al.* in the recrystallized volume behind the recrystallization interface. In the present investigation all of the major dissolution and reprecipitation of γ' particles appeared to be effected at the recrystallization interface: there was no evidence of persistent reprecipitation within the recrystallized volume.

The fact that reprecipitation was found to occur at the recrystallization interface emphasises the similarity of this process (mechanistically as well as morphologically) to that of a discontinuous precipitation reaction. However, the difference between the reprecipitation observed in the present investigation and a true discontinuous precipitation process is that in the present case the high solute supersaturation which results in precipitation is only likely to exist at or in the immediate vicinity of the reaction front (i.e. the recrystallization interface). This behaviour should be contrasted with that at the undissolved precipitates where high local solute supersaturations are dissipated as a result of rapid coarsening rather than reprecipitation.

The type of interaction that occurs between a recrystallization interface and a γ' particle distribution appears therefore to depend on a combination of factors. The most important of these must be the volume fraction of γ' (high in the present case), the size and spacing of the γ' particles (large in the present case) and the local driving force for the migration of the recrystallization interface (for a given deformation, the stored energy will be less for a warm-working than a cold-working process).

Both the coarsening and the reprecipitation processes found in the present investigation generate large γ' particles which tended to pin the recrystallization interfaces. These large particles have been found to determine the nature of the recrystallization behaviour which develops in the present alloy under conditions of static annealing following thermomechanical treatment [1].

4. Conclusions

The major effects of recrystallization on the γ' distributions in this material can be summarized as follows:

(a) The interaction between the recrystallizing interface and the original cuboidal γ' precipitate dispersion leads to a mixed microstructure containing fine, recrystallized γ grains and coarse γ' precipitates. The latter phase particles are of two types: (i) those which have their origin in the warm-worked structure and have coarsened at the recrystallizing interface, and (ii) those which have precipitated on the advancing interface. The existence of both these precipitate types (and especially Type ii) suggests that the solubility of γ' elements in the mobile boundary is extremely high, since the vast majority of impinging cuboidal γ' is dissolved in providing solute for their growth.

(b) The nature of the interaction of the recrystallizing interface with the precipitate dispersion can be modified by local conditions, which may promote more complex events, such as twinning.

(c) The γ' precipitates in the warm-worked regions have a cube/cube orientation relationship with the deformed matrix. In general, γ' particles coarsened at the recrystallization interface only have a close orientation relationship with the warm-worked regions which are immediately adjacent. This is a result of the high lattice curvature found in the warm-worked regions.

(d) All changes to the γ' particle distribution occur at the recrystallization interfaces: there was no reprecipitation *within* the recrystallized volume, other than fine spherical "cooling" γ'.

(e) The present results are in broad agreement with recent findings concerning recrystallization of a similar alloy after cold working [10].

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